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(54) METHOD FOR THE MANUFACTURE OF CAROTENOID PREPARATION

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(56) References Cited:
Japanese Kokoku Patent No. Sho 36[1961]-21476
Japanese Kokoku Patent No. Sho 37[1962]-8532
Japanese Kokoku Patent No. Sho 48[1973]-13687

(57) CLAIM

A method for the manufacture of a carotenoid preparation, characterized by adding more than 20 parts of a suspension oil to 80 parts of carotenoid with or without the subsequent addition of a surfactant, milling at a temperature at which the carotenoid is not eluted to a carotenoid suspension particle diameter below 1 μ, then uniformly dispersing in water.

DETAILED EXPLANATION OF THE INVENTION

The present invention concerns a method for the manufacture of carotenoid preparations, and its objective is the industrially advantageous manufacture of carotenoid preparations with an excellent coloration effect, i.e., dispersion effect.

Carotenoids are used as colorants. In general, carotenoids are manufactured as solids, which are dry hard lumps. Even when pulverized to a fine state for increased coloration effects, they are still hard, thus achieving the objectives is difficult. Commercial carotenoid powders currently on the market have a minimum diameter above 2 μ. They are not likely to fade, but the coloration is poor. To maximize the coloration effects, carotenoids are used as solutions in solvents (e.g., orange oil and other natural essential oils, hydrogenated limonene dimer, vegetable oils, etc.) for enhanced coloration effects. Such methods are described in Japanese Kokoku Patent Nos. Sho 48[1973]-13687, Sho 36[1961]-21476, and Sho 37[1962]-8532. However, such methods have the drawback of an extreme ease of fading.

One reason is that carotenoids are dispersed in molecular form in solvents.

Here, means for obtaining carotenoid microparticles without the solution method and carotenoid preparations that can be used easily have been problems to be solved.

Therefore, the development of industrially excellent manufacturing methods with excellent coloration effects and small changes with the elapse of time is desired. The present invention was made according to such desires. Next, the present invention is explained in detail.

First, in the present invention, the raw materials are carotenoids, such as carotene, bixin, ethyl-or methylbixin, lutein crypto, zeaxanthin, or their hydroxy or carboxy containing esters. They may be used alone or as mixtures thereof.

The suspension oils are fish oil, whale oil, cottonseed oil, rapeseed oil, sesame oil, peanut oil, other plant and animal oils, and natural essential oils that are liquid at room temperature or that can be melted by heating, hydrogenated limonene oil dimer, SAIB (sucrose acetate isohexabutylate), etc. They may be used alone or as mixtures thereof. Any amount can be used. However, in many instances, the amount used is more than 20 parts to 80 parts (by weight, same hereafter) of the carotenoid, while 190-400 parts is the range providing good results.

Next, surfactants such as glycerin fatty acid esters, sucrose fatty acid esters, sorbitan fatty acid esters, soya lipids, propylene glycol fatty acid esters, other nonionic surfactants, anionic surfactants, cationic surfactants, amphoteric surfactants, etc., are used. They may be used alone or as mixtures thereof. However, the use of surfactants is not essential.

Water is used as the dispersion liquid in any amount, while good results are obtained when used in an amount of 3-5 times that of the combination of carotenoids and oils.

Next, the manufacturing process is explained.

The carotenoid is added to a suspension oil and stirred evenly, then milled in the usual manner until the carotenoid particle diameter in oil becomes below 1 μ. A diameter below 1 μ means that more than half the particles has a diameter below 1 μ.

The milling has to be carried out in a mixed system of carotenoids and suspension oil, which is characteristic of the present invention. It is very difficult to mill the carotenoid alone to

a particle diameter below 1 μ, because the carotenoids are very hard solids and the heat generated in milling causes deterioration of the carotenoids, leading to a greatly reduced color retention.

The resulting mixture is treated with a surfactant, if needed, which may be added during milling. If added, the milling effect and dispersion effect described later are enhanced.

The resulting mixture is dispersed in water in the usual manner (emulsification, etc.)

The resulting product is the desired carotenoid preparation.

Here, the objective of the present invention is achieved.

Next, important effects and action of the present invention are explained.

The desired carotenoid preparations have good color development and an excellent coloration effect without quality deterioration with the elapse of time. Reasons are described below.

① The dispersed carotenoid particles are very small, yet even at this diameter the quality is stable. Conventionally, milling is done to make the carotenoid particles small, but obtaining small particles has been impossible because of agglomeration. We have learned that milling dispersion in liquid animal and plant oils enables the formation of small particles in the desired size. This effect is especially pronounced when a surfactant is added.

② In the carotenoid solutions in solvents, the carotenoids are dispersed in the form of a molecular dispersion, and we have learned that while carotenoids in a molecular dispersion state tend to fade in air and sunlight, fading with the elapse of time is very small with a particulate dispersion.

③ We have also learned that the smaller the carotenoid dispersion particles, the better the coloration effects, and the retention of such a small state can be attained only when the carotenoids in an oil are dispersed in water.

Such effects were obtained for the first time by the present invention.

The effects of the present invention are shown by the experimental examples below.

Effects of the coloration and fading of the present invention

Test item / Method	Coloration (coloring power)	Retention
Invention	80%	80%
Solution (conventional)	100%	4%

EXPERIMENTAL CONDITIONS

A sample consisting of 0.2 w/w% of that obtained according to the method of Application Example 1, 13 w/w% of sugar, 0.25 w/w% of citric acid, and 86.55 w/w% of water

was placed in a container (transparency above 80% at a wavelength above 360 mμ; liquid layer 4 cm) and irradiated for 3 h in a Fadeometer (UV carbon-arc light resistance tester; main wavelength in UV range of 380 mμ, energy on sample surface 38.3 mW•min/cm^2). Coloration and retention were visually evaluated. In the dissolution method (conventional method), 1 part of bixin is treated with 49 parts of vegetable oil, heated at 140°C for dissolution, mixed with 450 parts of a 20% gum arabic aqueous solution, and emulsified. A preparation was made with 1 w/w% of the product and 85.75 w/w% of water and tested similarly as above.

Next, the practical embodiments of the present invention are explained.

APPLICATION EXAMPLE 1

A mixture made from 1 part of bixin and 9 parts of a mixed oil made from 4.5 parts of SAIB and 4.5 parts of coco oil, as a suspension of coarse particles, was wet-milled (ball mill) until more than half the particles had a particle diameter below 1 μ.

The microparticle dispersion obtained was treated with 90 parts of a 10% gum arabic aqueous solution and emulsified to obtain the desired product.

This was added to a refreshment beverage and allowed to stand 10 days under sunlight. There was no discoloration and the coloration was bright.

APPLICATION EXAMPLE 2

Two parts of bixin were mixed with 9.5 parts of a mixed oil made from 5 parts of SAIB, 4 parts of coco oil, and 0.5 part of sorbitan trioleate to obtain a suspension of coarse particles, which was wet-milled until more than half the particles had a particle diameter below 1 μ. The microparticle dispersion thus obtained was mixed with 90 parts of a 12% gum arabic aqueous solution and emulsified using an emulsifying machine.

The desired product was obtained.

This product was added at a 0.1% concentration to a refreshment beverage. The coloration was bright, and no discoloration occurred when allowed to stand 1 month at room temperature.